


Design and Synthesis of 4-*O*-Podophyllotoxin Sulfamate Derivatives as Potential Cytotoxic Agents.

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Supplementary section

Page 2: ¹H-NMR of compound 1: Podophyllotoxin.

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PROTON NMR

compound 1 (podophyllotoxin)

2173.43
2128.47
1947.59
1905.90
1790.91
1789.81
1785.02
1432.92
1424.50
1416.12
1382.71
1375.55
1371.14
1367.46
1228.05
1218.52
1209.10
1137.17
1131.51
1120.52
1110.49
856.15
841.91
837.60
833.10
826.66
823.98
647.31
639.18
476.31
-6.75

Current Data Parameters
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EXPNO 591
PROCNO 1

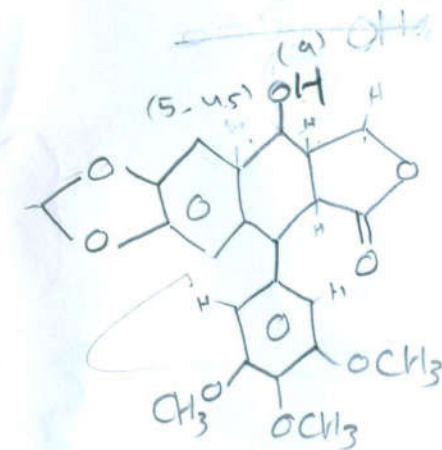
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PULPROG zg
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 5787.037 Hz
FIDRES 0.088303 Hz
AQ 5.6623602 se
RG 1290.2
DW 86.400 us
DE 5.50 us
TE 298.2 K
D1 1.00000000 se
MCREST 0.00000000 se
MCWRK 0.01500000 se

===== CHANNEL f1 =====
NUC1 1H
P1 8.60 us
PL1 -3.20 dB
SF01 300.1325269 MHz

F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.40 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.00 cm
CY 0.00 cm
F1P 18.020 pp
F1 5408.21 Hz
F2P -1.262 pp
F2 -378.83 Hz
PPMCM 0.83834 pp
HZCM 251.61029 Hz

Hz

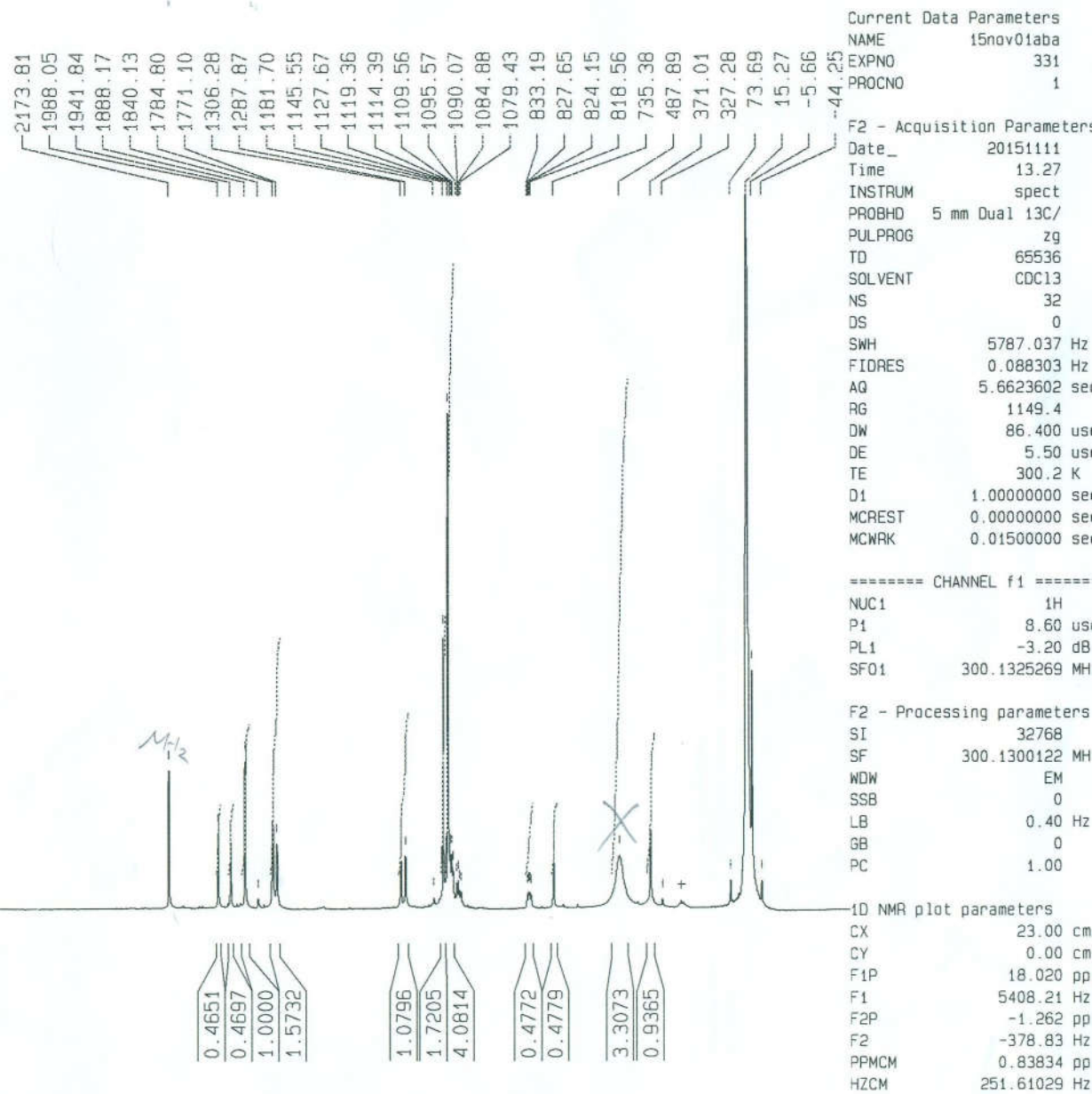
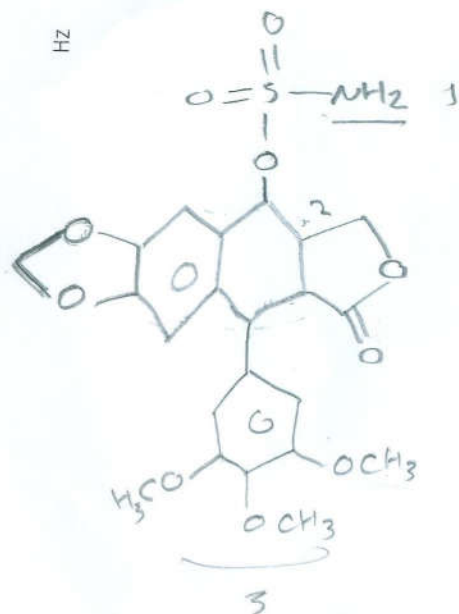


Integral

1.0000
1.9440
1.9158
1.0250
2.0201
0.9976
3.1911
5.7947
1.9245
0.9764

Compound 2; (4-O-Podophyllotoxin sulfamate)

PROTON NMR

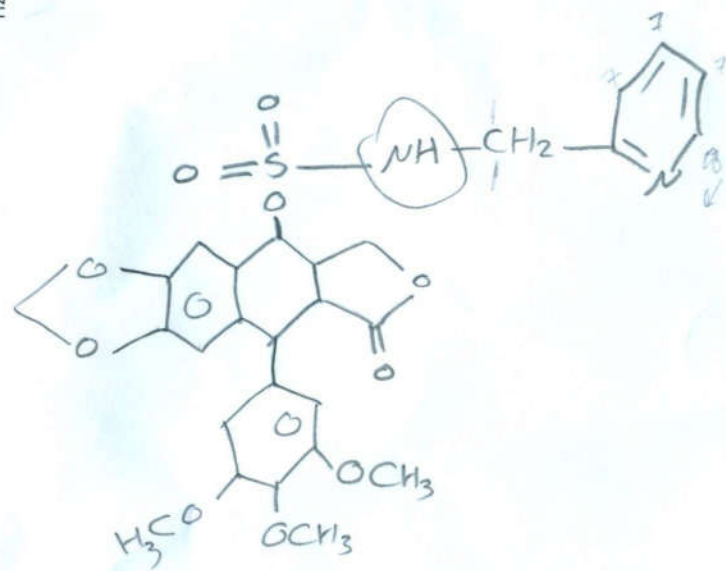


Integral

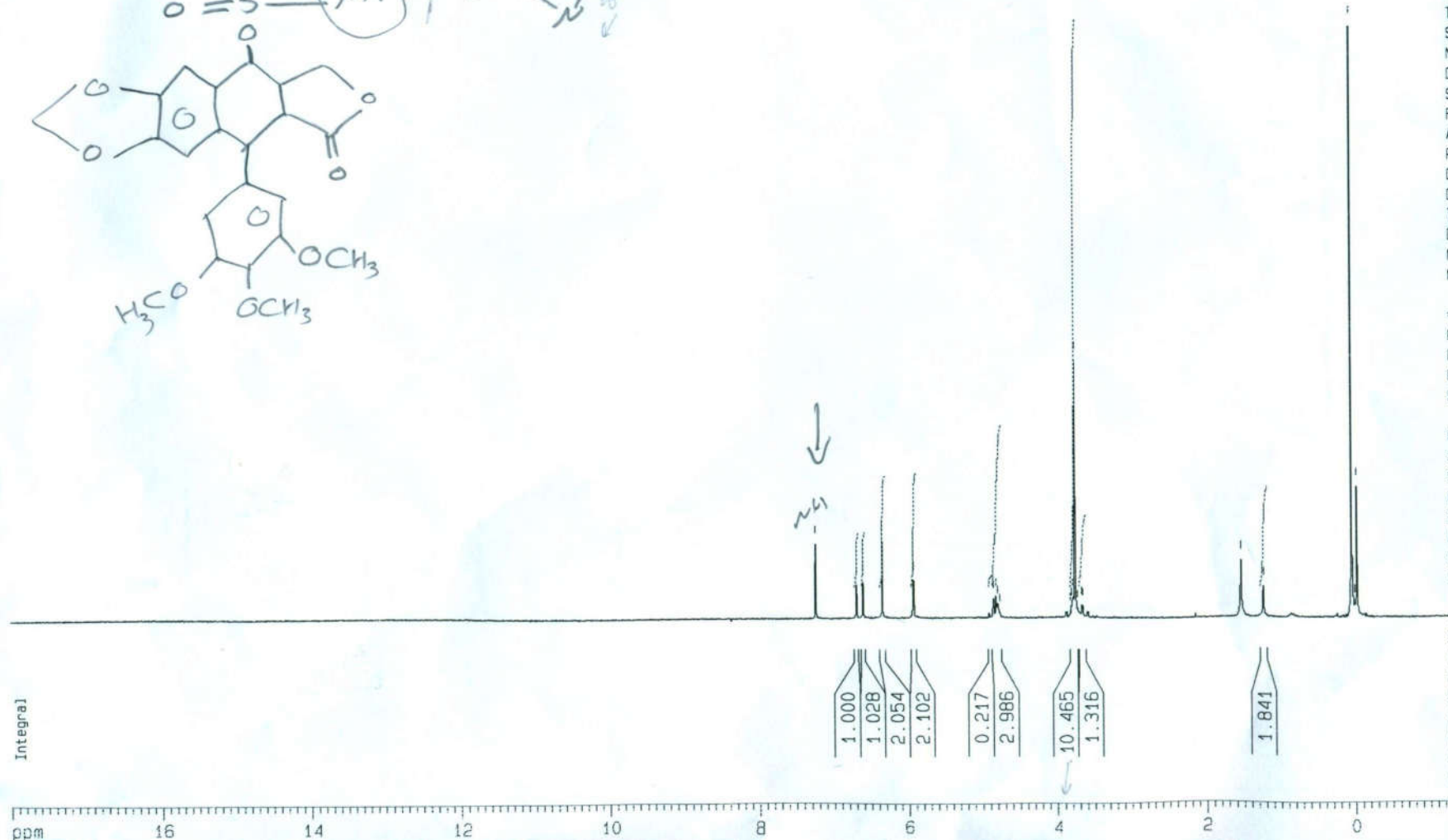
Compound 3: (4-*N*-(2-Pyridinylmethyl)-podophyllotoxin sulfamate)

PROTON NMR

Hz



2173.50
2011.42
1985.01
1907.14
1781.37
1779.50
1456.03
1445.76
1439.41
1430.71
1148.59
1142.80
1138.04
1131.82
1129.00
1120.59
1102.16
1098.47
461.65
370.76
15.05
0.50
-5.85



Current Data Parameters
NAME 15nov01aba
EXPNO 581
PROCNO 1

F2 - Acquisition Parameters
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Time 12.50
INSTRUM spect
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PULPROG zg
TD 65536
SOLVENT CDCl3
NS 1
DS 0
SWH 5787.037 Hz
FIDRES 0.088303 Hz
AQ 5.6623602 sec
RG 2298.8
DW 86.400 us
DE 5.50 us
TE 298.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

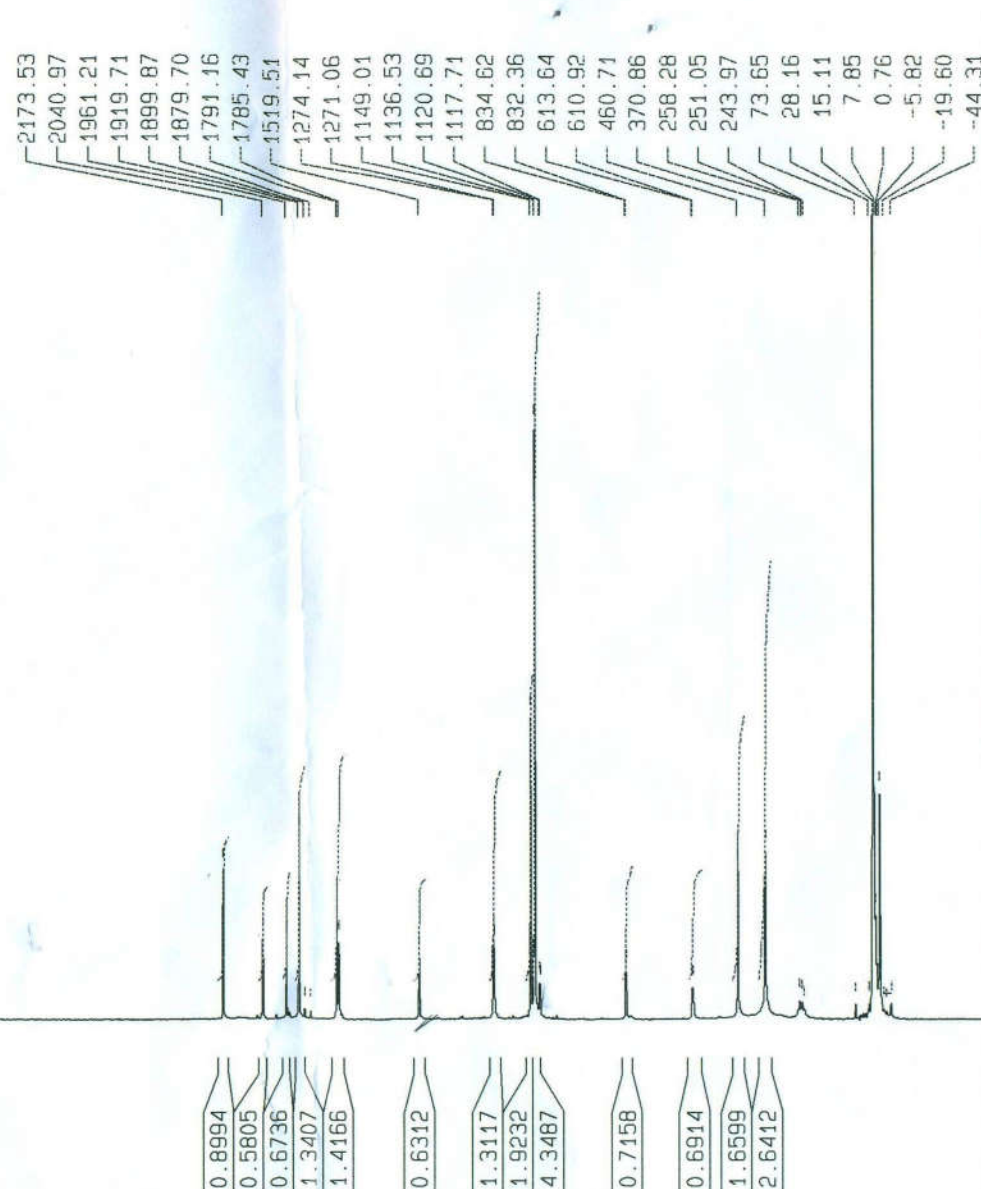
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NUC1 1H
P1 8.60 us
PL1 -3.20 dB
SF01 300.1325269 MHz

F2 - Processing parameters
SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.40 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 23.00 cm
CY 0.00 cm
F1P 18.020 ppm
F1 5408.21 Hz
F2P -1.262 ppm
F2 -378.83 Hz
PPMCM 0.83834 ppm
HZCM 251.61029 Hz

Compound 4: (4-N-(2-Pyridinylethyl)-podophyllotoxin sulfamate)

PROTON NMR



Current Data Parameters
NAME 15nov15aba
EXPNO 821
PROCNO 1

F2 - Acquisition Parameters

Date_ 20151123
Time 12.28
INSTRUM spect
PROBHD 5 mm Dual 13C/
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 6
DS 0
SWH 5787.037 Hz
FIDRES 0.088303 Hz
AQ 5.6623602 s
RG 1448.2
DW 86.400 us
DE 5.50 us
TE 298.2 K
D1 1.0000000 s
MCREST 0.0000000 s
MCWRK 0.0150000 s

===== CHANNEL f1 =====

NUC1 1H
P1 8.60 us
PL1 -3.20 dB
SF01 300.1325269 MHz

F2 - Processing parameters

SI 32768
SF 300.1300122 MHz
WDW EM
SSB 0
LB 0.40 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 23.00 cm
CY 0.00 cm
F1P 18.020 pp
F1 5408.21 Hz
F2P -1.262 pp
F2 -378.83 Hz
PPMCM 0.83834 pp
HZCM 251.61029 Hz

Hz

Integral



Compound 5: (4-N-(2-Pyridinyl)-podophyllotoxin sulfamate)

L4-S2
HNMR



The University of Jordan
Faculty of Science
Department of Chemistry

Instrument Model:
Bruker 500 MHz-Avance III

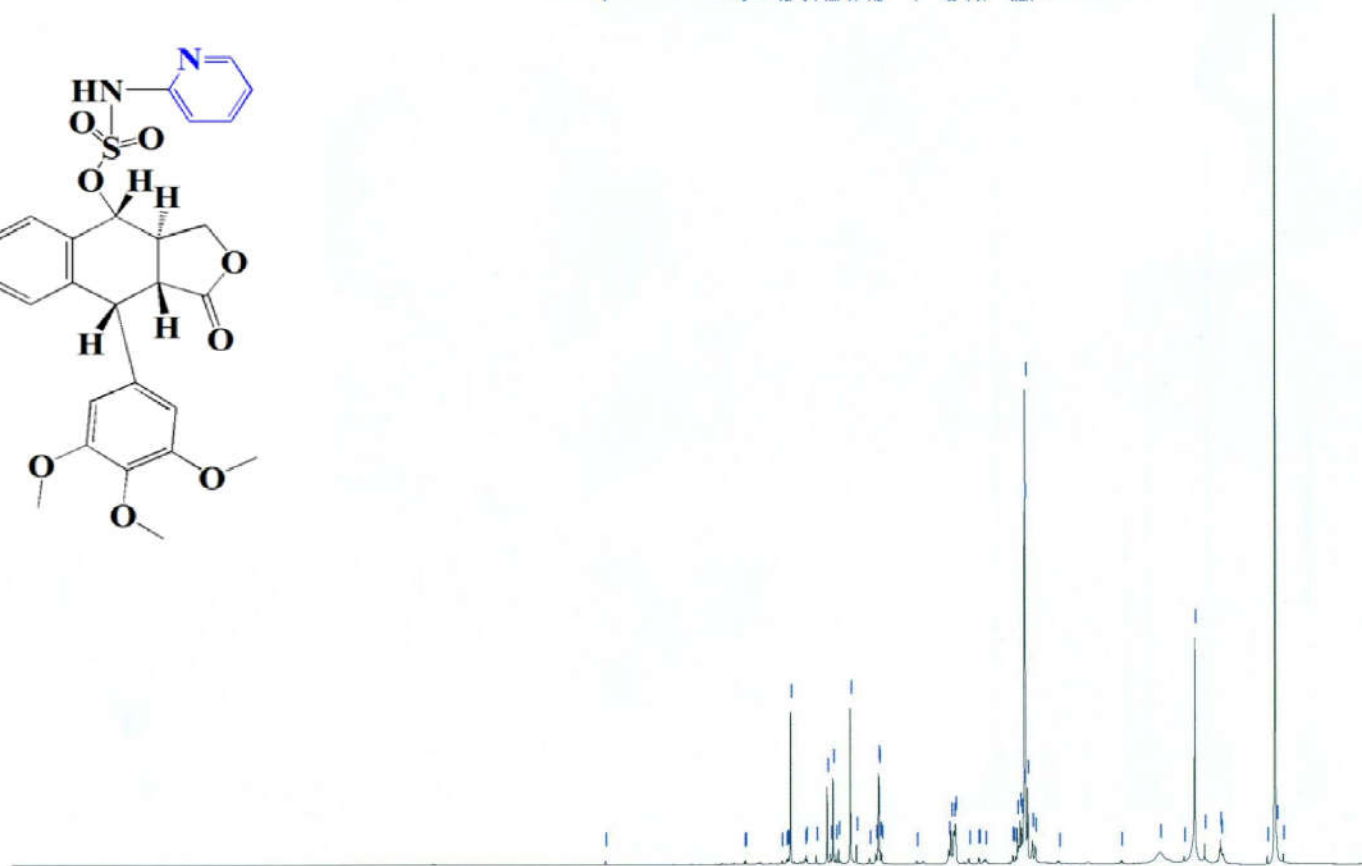
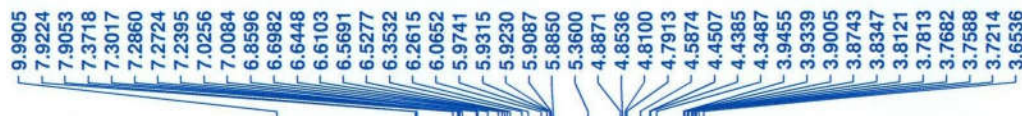
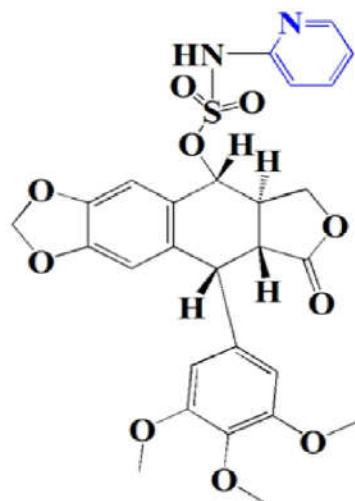
Operator: Rola Hassouneh
nmr500@ju.edu.jo

Current Data Parameters
NAME 15dec15jalal
EXPNO 541
PROCNO 1

F2 - Acquisition Parameters
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Time 8.47
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PULPROG zg
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 10135.135 Hz
FIDRES 0.154650 Hz
AQ 3.2331092 sec
RG 49.66
DW 49.333 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
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NUC1 1H
P1 10.60 usec
PLW1 17.39999962 W

F2 - Processing parameters
SI 131072
SF 500.1300235 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.50



18 17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm

1.00 0.39 0.79 2.57 1.89 1.63 1.18 2.76 5.17 1.38 1.50 1.80 8.36 4.10 25.02 3.60 24.15 3.92 4.95 18.09 6.19 3.00 4.96 36.53 5.28 2.08 1.83 3.49 12.59 21.68 19.44 3.80 4.21 5.20 9.35 12.17 28.95 23.83 37.62

L5-S2
HNMR

Compound 6: (4-N-(4-fluorophenyl)-podophyllotoxin sulfamate)



The University of Jordan
Faculty of Science
Department of Chemistry

Instrument Model:
Bruker 500 MHz-Avance III

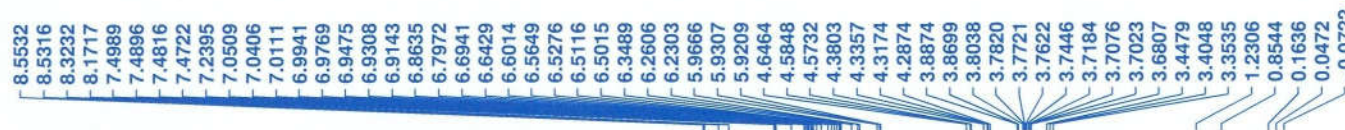
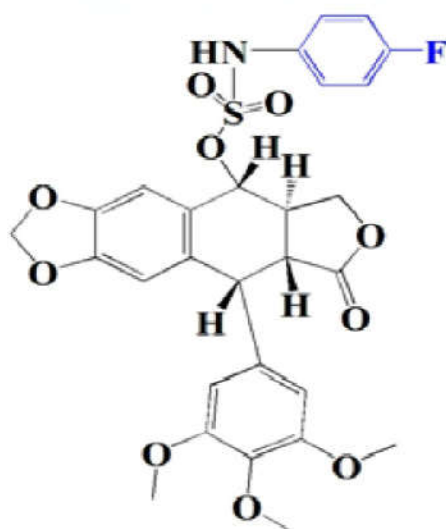
Operator: Rola Hassouneh
nmr500@ju.edu.jo

Current Data Parameters
NAME 15dec15jalal
EXPNO 511
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151231
Time 9.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg
TD 65536
SOLVENT CDC13
NS 32
DS 0
SWH 10135.135 Hz
FIDRES 0.154650 Hz
AQ 3.2331092 sec
RG 35.17
DW 49.333 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1344108 MHz
NUC1 1H
P1 10.60 usec
PLW1 17.39999962 W

F2 - Processing parameters
SI 131072
SF 500.1300235 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.50



18 17 16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 ppm

1.00 1.47 0.91 4.22 3.86 3.09 0.55 0.18 0.22 0.13 0.37 0.11 0.21 0.15 0.32 0.29 0.49 0.29 0.25 0.99 0.45 1.34 0.41 0.73 1.40 1.06 0.42 0.44 0.31 1.81

Compound 7: (4-N-(2-Anthracenyl)-podophyllotoxinsulfamate)

L6
HNMR



The University of Jordan
Faculty of Science
Department of Chemistry

Instrument Model:
Bruker 500 MHz-Avance III

Operator: Rola Hassouneh
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Current Data Parameters
NAME 16jan15jalal
EXPNO 131
PROCNO 1

F2 - Acquisition Parameters
Date_ 20160118
Time 12.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 10135.135 Hz
FIDRES 0.154650 Hz
AQ 3.2331092 sec
RG 71.89
DW 49.333 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
SFO1 500.1344108 MHz
NUC1 1H
P1 10.60 usec
PLW1 17.39999962 W

F2 - Processing parameters
SI 131072
SF 500.1300235 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

